

Gas Chromatographic Instrumentation for Gas Analysis of the Martian Atmosphere

FINAL REPORT

Volume I

Third Monthly Letter Technical Report (Final)

GPO PRICE \$ _____

OTS PRICE(S) \$ _____

Hard copy (HC) 2.00

Microfiche (MF) .50

25 September 1962

Contract No. 950326

Submitted to

JET PROPULSION LABORATORIES
Pasadena, California

MELPAR INC

A SUBSIDIARY OF WESTINGHOUSE AIR BRAKE COMPANY

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FACILITY FORM 802

N 65 12615

(ACCESSION NUMBER)

34

(PAGES)

CR 59769

(NASA CR OR TMX OR AD NUMBER)

(THRU)

1

(CATEGORY)

14

RC. # 62-432

GAS CHROMATOGRAPHIC INSTRUMENTATION
FOR
GAS ANALYSIS OF THE MARTIAN ATMOSPHERE

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Third Monthly Letter Technical Report

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Submitted to
Jet Propulsion Laboratories
Pasadena, California

Submitted by
Melpar, Incorporated
3000 Arlington Boulevard
Falls Church, Virginia

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1. INTRODUCTION

In compliance with the terms of JPL Contract No. 950326, Melpar submits this final report on the Gas Chromatographic Instrumentation for the Analysis of the Martian Atmosphere. Under this contract a laboratory model of the desired unit has been constructed. The unit is now ready for demonstration.

The gas chromatographic system developed at Melpar consists of a series arrangement of columns with appropriately placed cross-section ionization detectors. Unpurified helium may be used as the carrier gas. Complete analyses with the present unit may be made in just under 5 minutes, and provisions have been made for carrying out at least four complete determinations. A certain amount of analytical redundancy incorporated into the system serves as a basis for checking the functionality of the unit.

The gas chromatographic system is highly reliable and quite stable. The unit is ready for operation within a few minutes after the electronics are energized and the carrier flow is started.

Since there are numerous facets of the effort to be considered in the final report, it seems advisable, for a clear presentation of the effort, to break this report into three distinct parts as follows:

- a. Volume I: Third Monthly Letter Report.
- b. Volume II: Overall Program Summary.
- c. Volume III: Post Design Criteria and Summary.

In Volume I, a summary of the technical effort for the last month is considered. This effort for the most part has been directed towards the integration of all components into a workable laboratory model. Volume II

summarizes the gas chromatography system as a laboratory model. Performance characteristics, significant technical data, and design drawings covering the overall unit and component parts are presented. In Volume III, the projected design considerations for the breadboard and prototype gas chromatography packages are covered. Trade-offs that logically could be made and certain recommendations are also considered.

Melpar considers it a pleasure and a privilege to work with JPL under this contract. Many formidable problems have been overcome thus far in the development of a functional gas chromatographic instrument. Much of the effort in this phase of the program has been directed toward the development of many components which would begin to meet the stringent requirements of the breadboard instrument. These components with minor modification, it is felt, would be adequate for the breadboard. Melpar's design considerations show that all components can be fabricated to meet the breadboard criteria and the overall packaging criteria of weight and volume can be met.

2. MECHANICAL DEVELOPMENT

2.1 Introduction

The mechanical development effort in this program has been directed toward: (1) the construction and evaluation of an optimum sample injection valve, (2) a carrier-gas control system, (3) provisions for instrument sealing, (4) provisions for loop switching, (5) development of a Martian atmospheric Venturi gas pump, (6) construction of a Martian-atmosphere simulator, (7) construction considerations for the laboratory model, and (8) design considerations for a breadboard model.

Previously the fabrication of two injector valves of alternate designs was carried out. The overall layout and construction of the laboratory model was initiated, and the availability of a carrier gas control system was investigated. In addition the initial design for the transit sealing of the instrument was considered.

During this period a third and final model of the injector valve has been designed and constructed. This valve incorporates the flat slider-type valving and squib firing actuation. The layout and mounting of the laboratory model has been completed, and a design for the carrier-gas control has been developed. The fabrication of a transit sealing device as well as a reference gas capsule device has been made. The packaging design for a breadboard model of the gas chromatographic unit was developed.

2.2 Experimental Procedure

The evaluation of the two previously fabricated injection valves has been made. The design based on the concept of using the carrier gas for pneumatic actuation of the valve without loss of the gas may not be feasible.

The pressure change at the interface of valve and column head is, with the present design, insufficient for adequate venting of the valve. The second design, embodying the slider valve, appeared to provide the optimum characteristics desired in the laboratory model. On the basis of this information, a third valve based on the slider principle was fabricated. This valve approached in size and weight a valve that would be acceptable for a bread-board model. This valve uses four squibs at each end of the slider valve to drive the valve from the sampling position to the inject position and from the inject position to the sampling position. Thus, four samplings using eight squib actuators may be realized.

Since the squib-actuated pistons once fired cannot readily be returned to their original positions, a small link was placed between the actuator and the cage which moved the slider on the valve. This device, after moving the cage, enabled the link to be rotated so that the cage could come back and pass it without interference.

Considerable effort has been expended in obtaining as near perfect a seal on the Teflon slider and stainless steel port block as feasible. (See the previous monthly report for a sketch of this valve). Pressure is maintained on this interface by means of a metal back up spring. The two surfaces are lapped as perfectly as possible. Tests were conducted on this valve using air pressure at 60 psi on the helium and column ports with a dummy loop across the loop ports and a high vacuum on the intake and exhaust ports. The vacuum drawn on these ports was in the order of 50 microns of mercury. Under these conditions, a minimum leakage of 25 microns of mercury per minute was observed when the system was isolated from the

vacuum pump. It is believed that a portion of this leakage may have arisen from the metal couplings and sealant used in the test setup. Adjustments were made in the valve to ensure that with actuation no appreciable dead volume pick-up was obtained from the closed port on the slider valve. This dead volume pick-up was observed to be of the same general magnitude as the leakage observed with the isolated system.

The use of Teflon in any application where mechanical accuracy is an important consideration always raises the question of its cold flow characteristics. This is particularly true in this application where the Teflon is under considerable pressure from the spring on one side and pressure and vacuum on the other side. A preliminary check of the cold flow affect was made by allowing the valve to remain in the spring-loaded condition overnight. The next morning the leak rate of the valve was compared with that of the previous evening. The same leak rate observed on the previous day was obtained.

This valve appears to be feasible for use in the laboratory model and has been so incorporated.

The carrier-gas control for the laboratory model, utilizing the previously described injector valve, will consist of a commercial helium tank supply and a standard pressure regulating valve which will feed directly into the injection valve. As was mentioned in the previous report, it was decided to forgo the purchase of the titanium tank and pressure regulator for the laboratory model for economic reasons. Both of these items, which are readily available, will of course be further discussed and considered in the breadboard design evaluation.

A column sealing system has been fabricated. (This same type of device can be used for the carrier-gas tank sealing). This arrangement consists of a gas-squib actuator and, in the present model, a glass diaphragm seal. This system will serve to illustrate the principle of using a diaphragm ruptured by a gas-squib actuator for sealing either the columns or the carrier-gas tank.

A laboratory model reference gas capsule device has also been fabricated. This device consists of a T-connection with a glass ampule placed between glass-wool filters and ruptured at the appropriate time by a squib actuator. This device can be incorporated either into the injector valve or the line to the columns from the helium tanks as required.

A chassis was fabricated for the mounting of the laboratory model at the beginning of this period. The overall system was assembled on this chassis. Accessibility of the various components in the test columns and detectors was emphasized in this model in order to allow conduction of the experimental work on the gas chromatograph as integration continued. Although changes in components have resulted in a considerable variation from the original design, the general configuration remains the same.

A Venturi pneumatic pump has been designed, fabricated, and evaluated for use with the Martian gas chromatograph. In this pump the flow of exit carrier gas is used to charge the sample loop with atmospheric sample. The device consists of a critical orifice at the downstream end of the columns of the chromatograph followed by a Venturi tube. Experimental work using an eight to ten one-thousandth-inch orifice has indicated that a flow rate of 400 milliliter per minute through the critical orifice will

provide approximately 1.6 liters of circulation of the Martian atmosphere gases. Determinations of parameters of this device were obtained on an empirical basis rather than a theoretical basis because of the large variations in the constants involved. This device appears to provide an excellent means for circulating the Martian-atmosphere gases through the instrument after impact.

In figure 1 is given a schematic showing the above components of the system.

2.3 Results

Design, development, and fabrication of the various components have been completed. The laboratory model chassis has been constructed and integration of the system has been completed. It is apparent from the work done during this period that a breadboard design will evolve which will meet weight and volume requirements. The unit should be compatible with the extreme environmental conditions imposed upon it during space and travel entry into the Martian atmosphere.

2.4 Work for the Next Month

The only remaining work to be done is completion of the other aspects of final report including the recommended breadboard design for the gas chromatograph. This work will be completed.

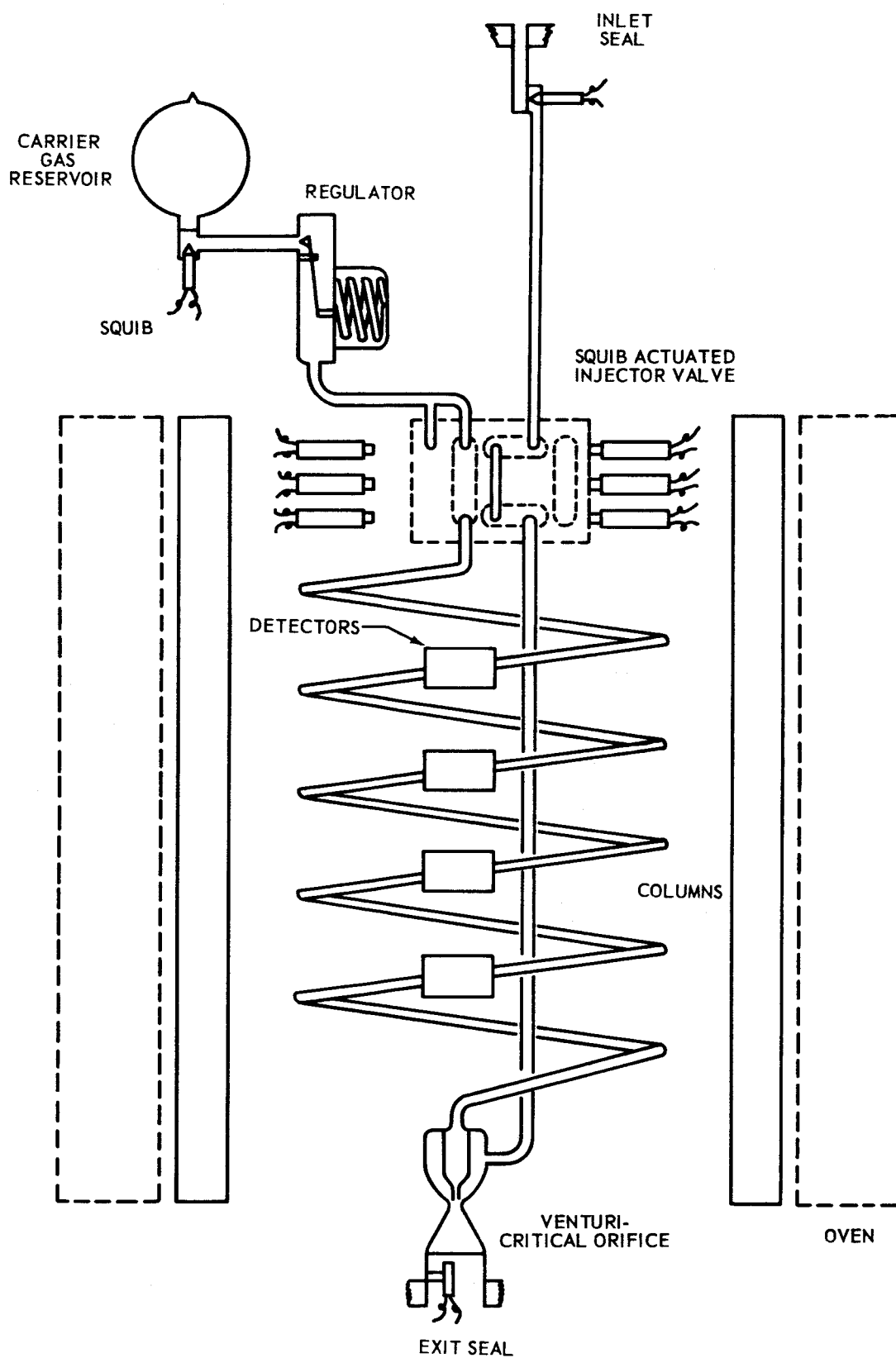


Figure 1. Schematic of the Overall Layout

3. COLUMNS

Primary emphasis during this phase was placed on integration of the four columns into a series arrangement. The arrangement that finally evolved is shown in figure 2. This system was designed to give a reasonable compromise between efficiency of separation, temperature requirement, and gas requirement when a 5-minute analysis period is used. All results presented in this report were made with this system operating at 27°C and a flow rate of 175 ml He/min STP. Absolute helium pressure at the injection valve was 80 psi while that at the exit was 20 psi.

Typical chromatograms, as seen by amplifiers 1 and 2, are shown in figures 3 and 4, respectively. It may be seen that essentially complete separation exists between all compounds shown, with the exception of the ethane-N₂O pair. H₂S is not resolved from the air peak on the liquid column and it is not eluted from the silica gel column.

The first column, the liquid column, was designed to elute NH₃ shortly after air and to elute water within 5 to 6 minutes. This elution sequence has been accomplished. As reported previously, more polar liquid phases would reduce NH₃ tailing, but these also would increase the retention of water. Basic liquid phases also would reduce NH₃ adsorption, but such phases have been found to be extremely unreliable over a period of time.

Column 2A is simply an empty tube designed to delay the entry of air into detector 2 until NH₃ has been eluted from detector 1. Column 2B is a short silica gel column used in attempting to separate Xe, N₂O, C₂H₅, and CO₂. We have been unable to obtain any separation of CO₂ and N₂O from each other or silica gel columns. Also, with the short column necessary,

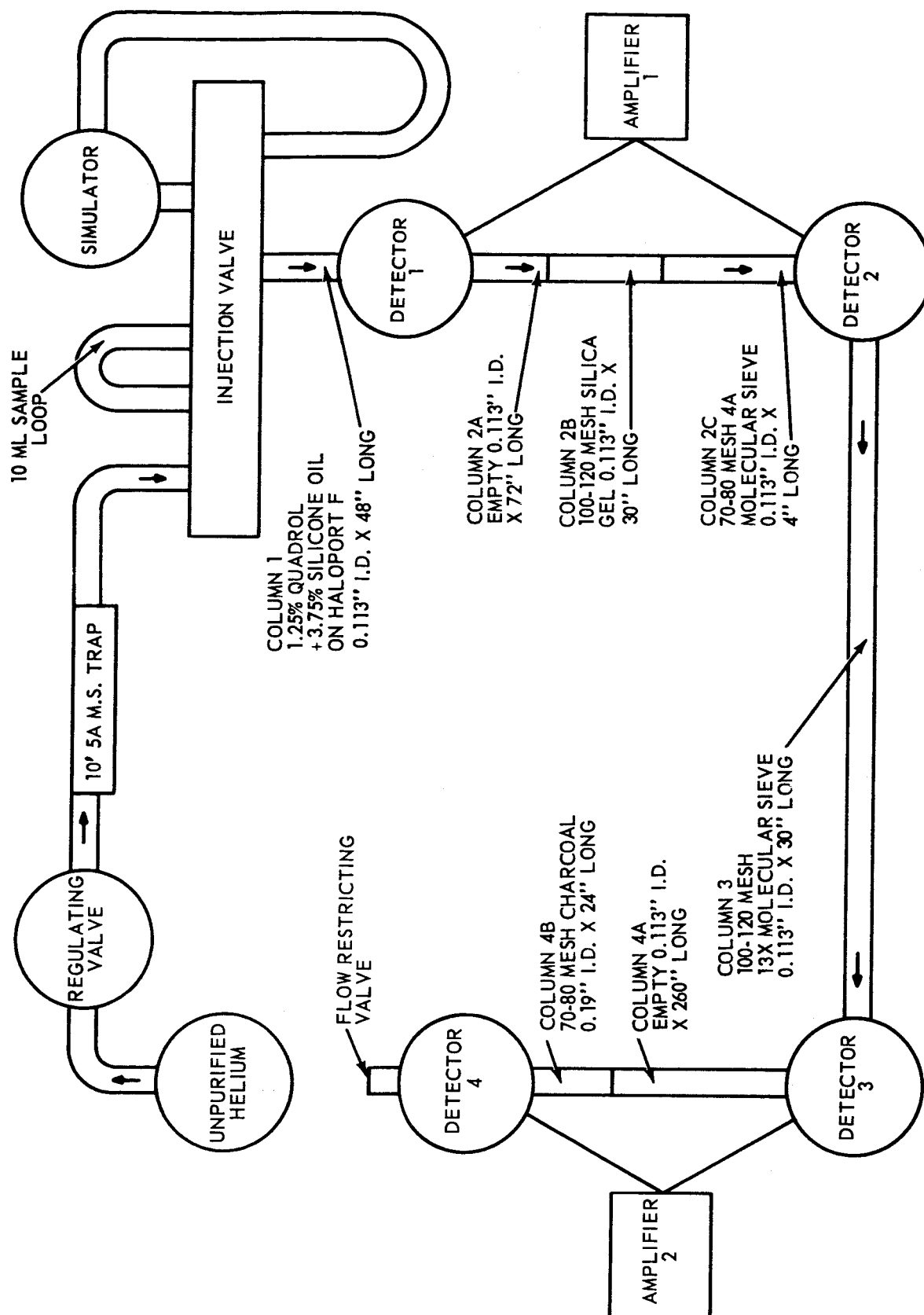


Figure 2. Schematic Diagram of the Gas Chromatographic System

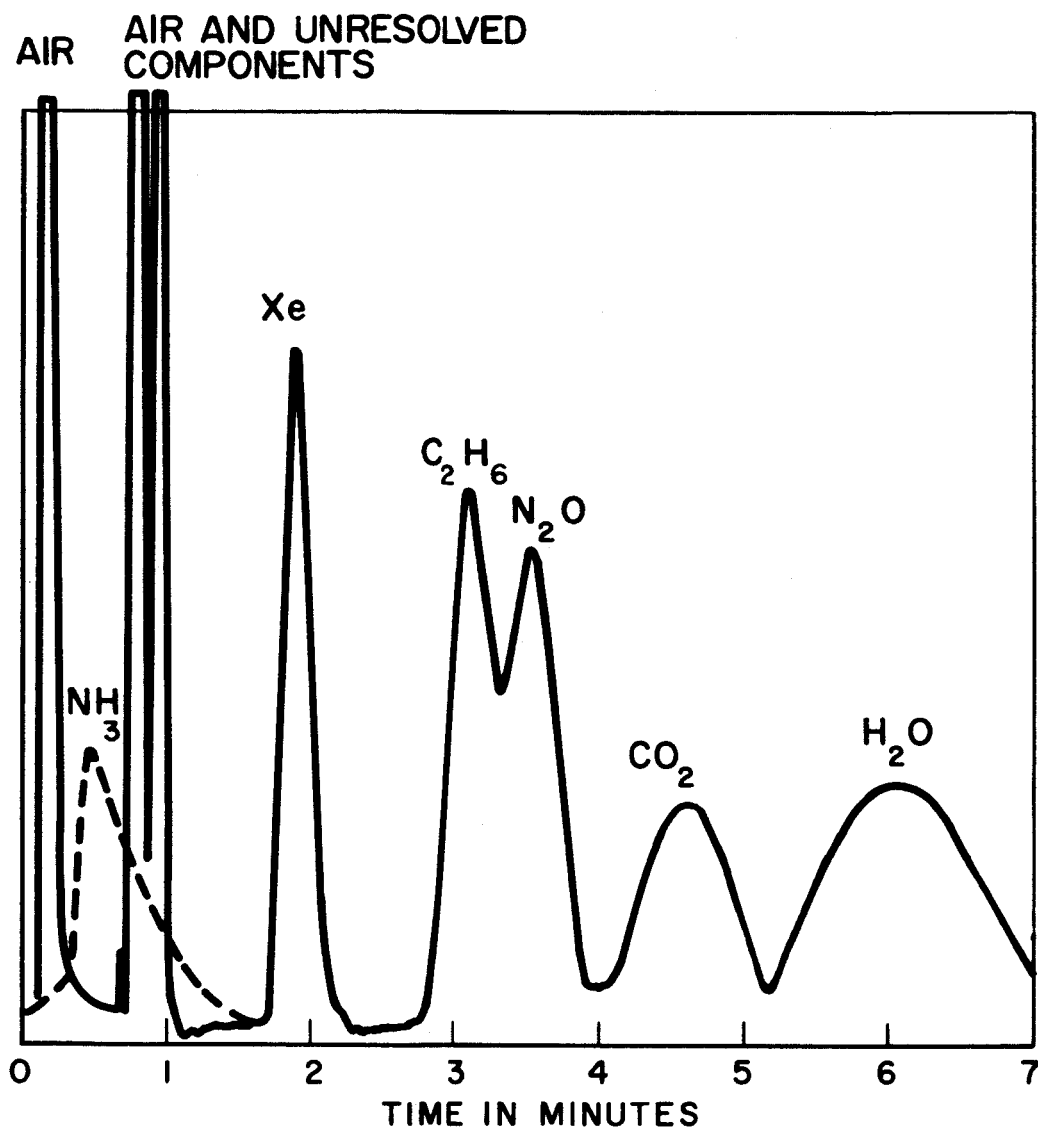


Figure 3. Typical Amplifier 1 Chromatogram

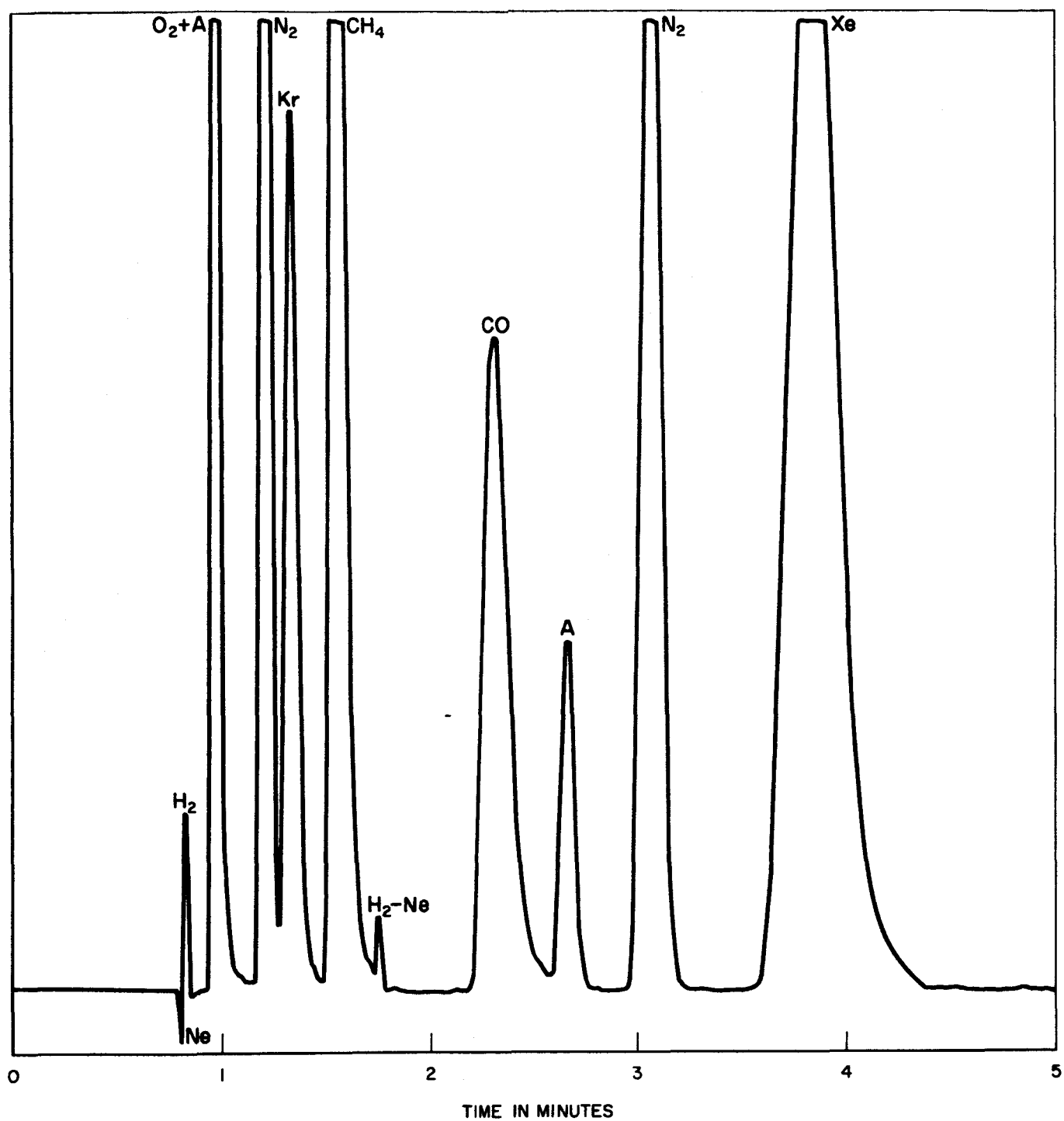


Figure 4. Typical Amplifier 2 Chromatogram

ethane - CO₂ separation has been very poor. Column 2C, a very short molecular-sieve column, corrects these difficulties by retarding the CO₂. This short column also allows complete separation of CO₂ from N₂O to be achieved. Although this column does broaden the CO₂ peak, it is believed that the advantages gained by the N₂O separation far outweigh this slight disadvantage. NO and NO₂ are not passed by these columns.

Column 3 is a conventional molecular-sieve column. The excellent separation obtained between N₂ and Kr is associated with the silica gel column which precedes this column rather than to the molecular-sieve column itself. It may be noted that Xe also is eluted from column 3, thus giving a qualitative and quantitative check on the performance of columns 2 and 3 and detectors 2 and 3, respectively. Hydrogen and neon are not separated; but this disadvantage is overcome to a large extent by the detector characteristics to be discussed shortly. Argon and oxygen are not separated here but this shortcoming is overcome by column 4.

Column 4A is a delay column designed to prevent interference of compounds seen by detector 3 with those seen by detector 4. Column 4B is filled with highly activated charcoal to completely adsorb oxygen. It is constructed from larger tubing in order to reduce the carrier-gas velocity and thus increase its efficiency. It may be seen that hydrogen, argon, and nitrogen eluted from this column do not interfere with compounds eluted from column 3. The repetition of the hydrogen and nitrogen peaks give a further check on the functionality of the system. Oxygen is not eluted unless over 20 ul are injected. It then is eluted as a broad peak after nitrogen. This column must be reconditioned after approximately 200 hours

of operation since water will tend to inactivate it, thus causing O₂ and A peaks to be eluted together. This would not be a problem for the Martian application.

This column system, while not completely optimized, is believed to be entirely adequate for the Martian analysis. With this system, efficiency, analysis time, and relative retention times can be altered by changing the carrier-gas flow characteristics. Higher inlet pressures have been found to improve the efficiency and speed of analysis. Efficiency improvement is most noticeable on the first columns, whereas speed improvement is most noticeable on the last columns. Higher outlet pressures improve the efficiency and reduce the speed of analysis. Both efficiency improvement and speed reduction are most noticeable on the last columns. Consequently, if both inlet and outlet pressures are increased so that the speed of the last columns remain the same, the first columns will exhibit improved efficiency and speed and the last columns will exhibit improved efficiency. This, however, would require a heavier injection valve to prevent leakage and also would require a greater helium consumption.

4. DETECTORS

Microcross-section detectors were used exclusively in the last phase of this work. These detectors were quite appealing because of their extreme stability, reproducibility, and minimum activation time. Our experience with helium ionization detectors with which purified helium is used, has shown that they are temperamental and at times unreliable. Melpar doubts the applicability of helium ionization detector to this problem. There is no doubt that if sufficient pains were taken a system could be developed to perform properly here on earth. But even with a workable system here the question arises so to just how functional the same system would be after a transit time of some 7 or 8 months. Certainly any change in the impurity content in the helium from desorption or other sources could change markedly the response characteristics of the detector.

The microcross-section detectors normally operate in the plateau region of the current voltage curve, and consequently their response is independent of potential within a wide voltage range. It has been found that the voltage required to put the detector in the plateau region is directly proportional to the helium pressure within the cell. For the series column arrangement of figure 1, this voltage would diminish in going from detectors 1 to 4. To compensate somewhat for the pressure effect, the electrode spacing on the first detector was reduced compared with that on the others. (The potential necessary for attaining the plateau region varies approximately as the square of the electrode spacing). The first detector was constructed to give an electrode spacing of $1/16$ inch and the other three detectors were set at an electrode spacing of $1/8$ inch. All detectors used had 0.40-inch cell chamber diameters.

On the parallel plate electrodes of detectors 1 and 3 was mounted tritium foil supposedly having an activity of 1 curie/in². The tritium foil in detectors 2 and 4 supposedly had an activity of 4 curies/in². All detectors had base line currents of 1.5×10^{-8} amperes at atmospheric pressure irregardless of the tritium foil used. This common base current was not an anomaly of any kind. Rather, as confirmed by the source (U.S. Radium), the surface activity of the tritium foil purchased was approximately the same irregardless of the reported activity.

All detectors reached their current plateau at 11v or slightly less when operated at atmospheric pressure. However, the higher pressures within the series column arrangement necessitated somewhat higher voltages. Forty-five volts was found to be sufficient for detectors 1 (with its 1/16-inch spacing), 3, and 4. Detector 2, with a spacing of 1/8-inch and greater helium pressure, required a higher potential than did detectors 3 and 4. This detector performed quite satisfactorily at 135 volts. A 1/16-inch electrode separation in detector 2 would have enabled this detector to be operated also at 45 volts. The appropriate common voltage for all detectors will be considered in Volume III.

Detectors 1 and 2 are connected to amplifier 1 and detectors 3 and 4 are connected to amplifier 2 (see figure 2). When connected this way, the noise level from amplifier 1 was 3×10^{-12} amperes while that from amplifier 2 was 2×10^{-12} amperes. Drift during the 5-minute analysis time was insignificant.

The calibration of the system's detectors has been completed. Typical calibration data are shown in figures 5, 6, and 7 for water, xenon, and

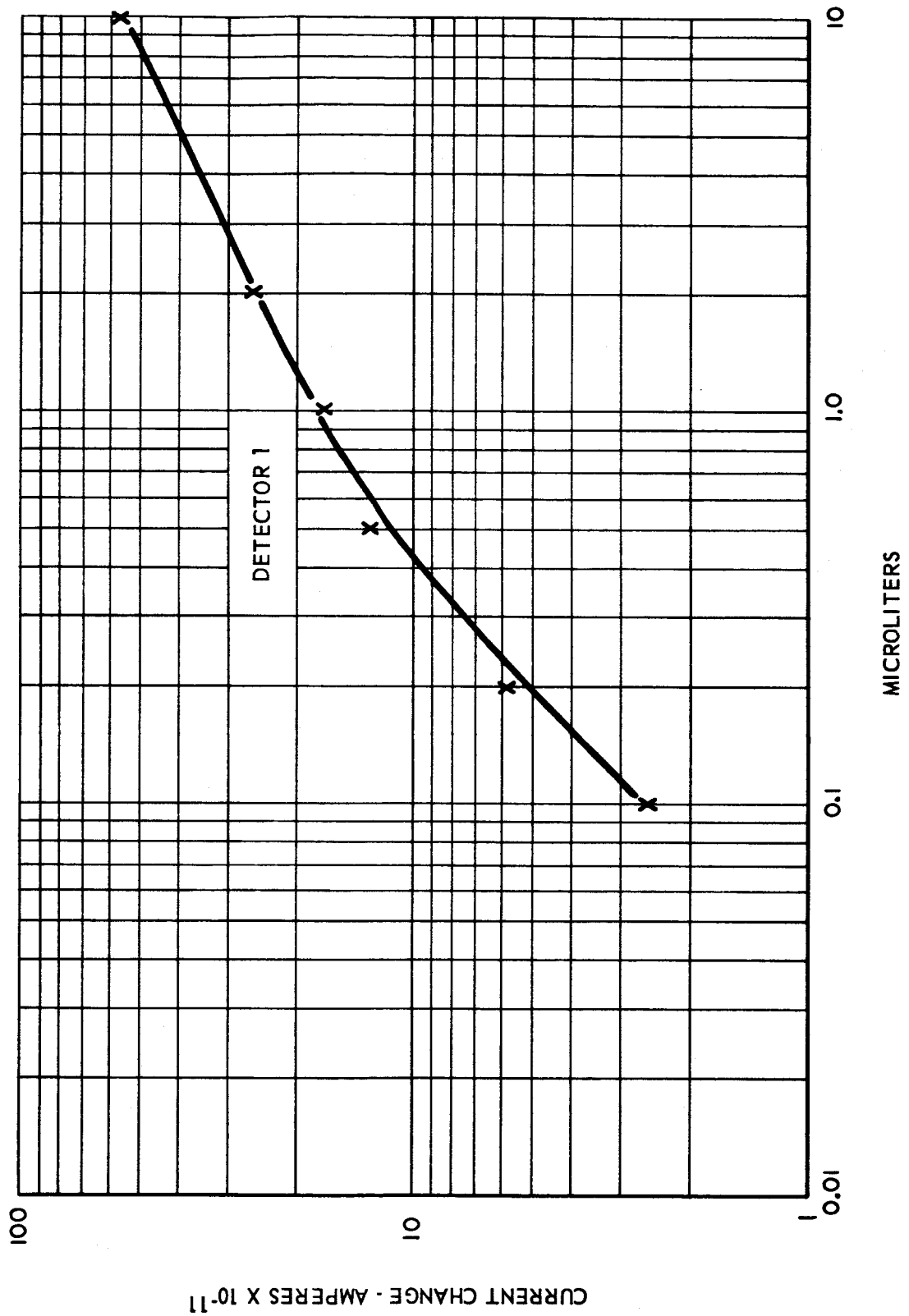


Figure 5. Response of Cross-Section Detector to Water

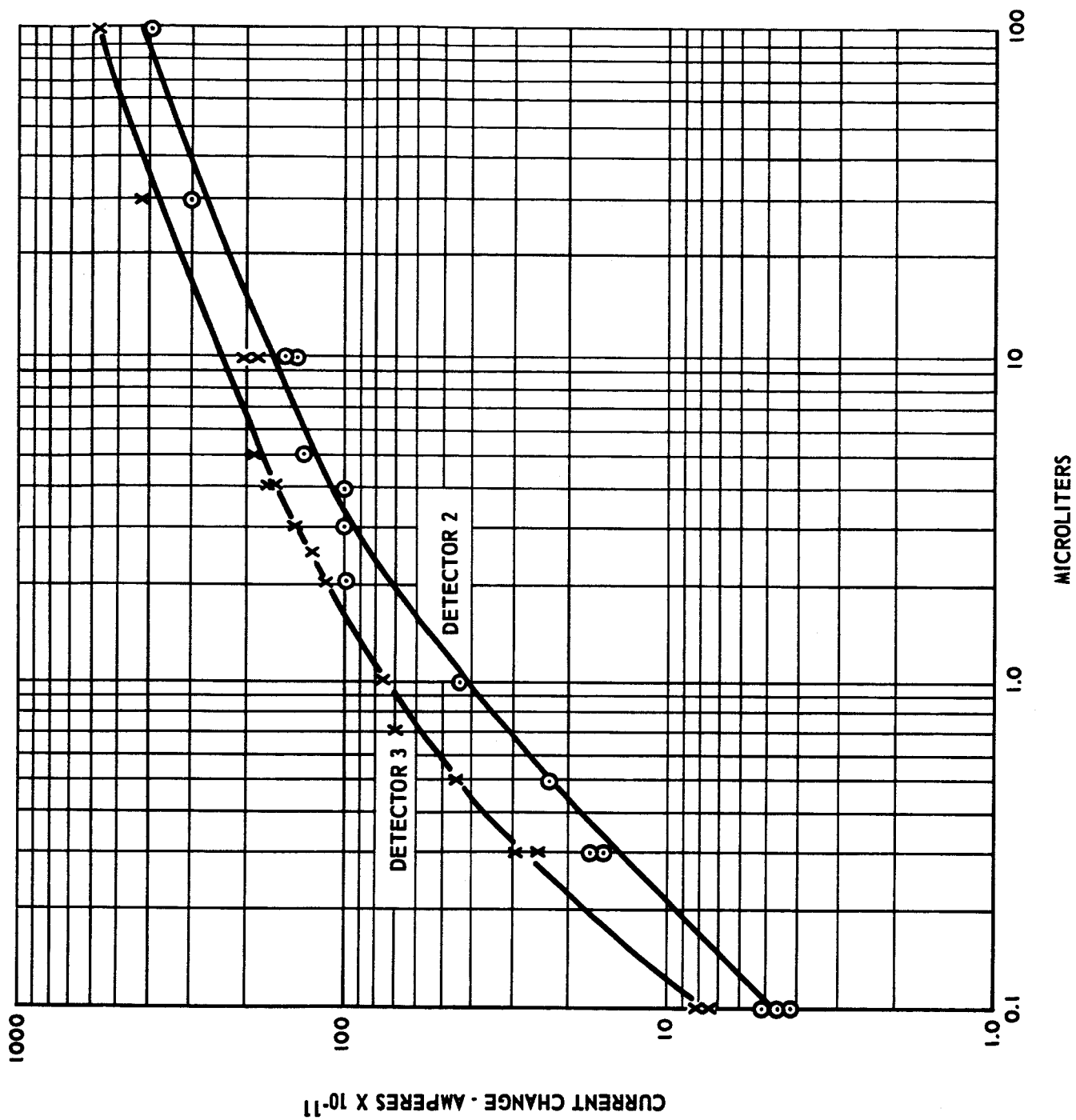


Figure 6. Response of Cross-Section Detector to Xenon

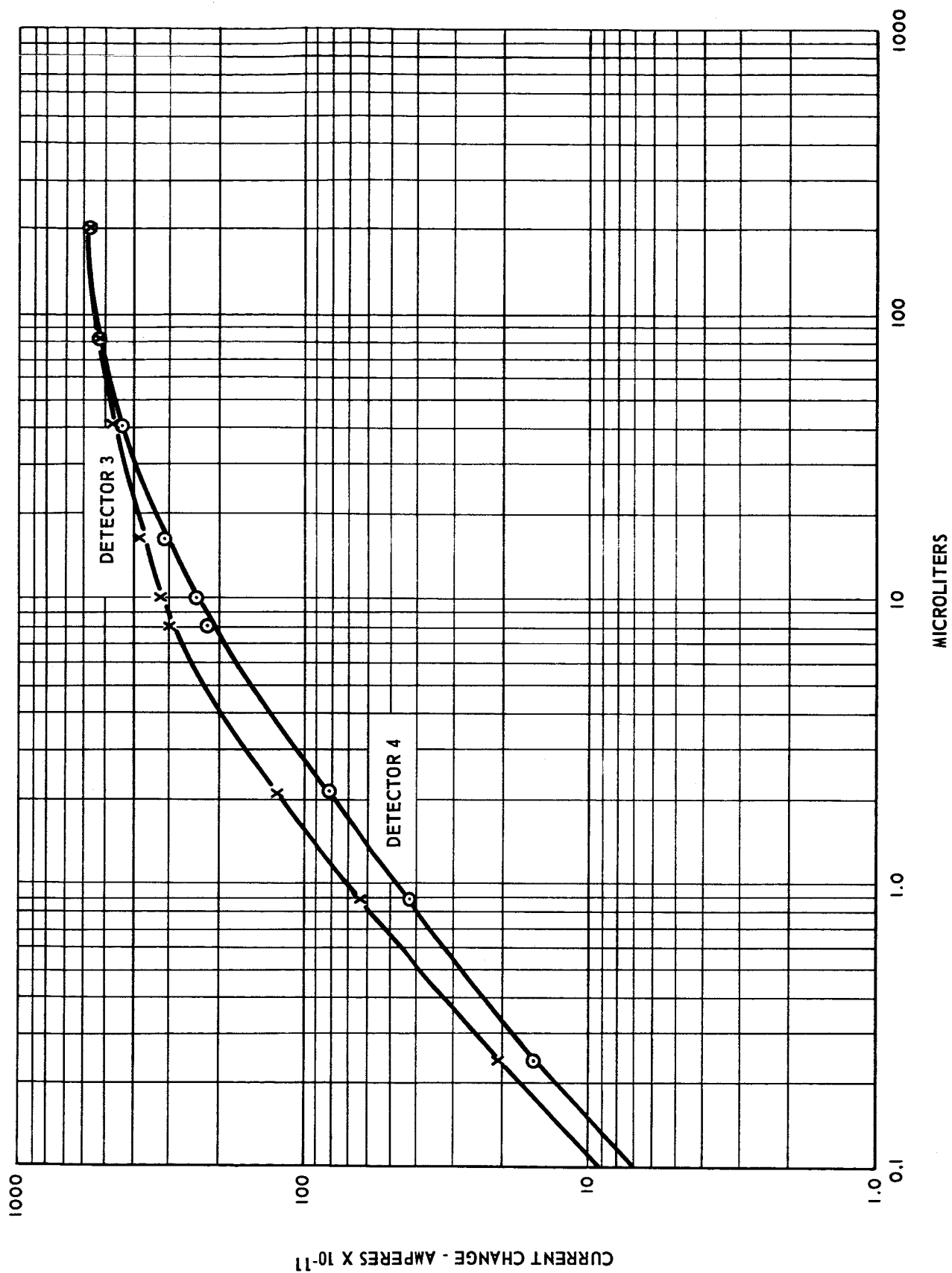


Figure 7. Response of Cross-Section Detector to Nitrogen

nitrogen. Detector numbers in these figures apply to figure 2. These data were obtained by syringe injection. (Contamination of the Martian-atmosphere simulator at the last minute precluded its use in conjunction with the sample valve. A new, all glass simulator has been constructed; this will be used to calibrate the entire system with the sample loop in position on the sample valve). These calibrations were obtained with the electrometers and automatic zero suppression circuits fabricated for this program incorporated into the system. More detailed calibration curves will be given in Volume II of the final report.

These results indicate that less than 0.01 ul of these compounds could be detected and they also indicate that the response is not limited by the sample size. The response characteristics of the detectors is slightly nonlinear; but this nonlinearity is used to advantage. The logarithmic amplifier previously considered for this program no longer appears to be necessary for use in conjunction with the amplifiers; that is, the dynamic range of the amplifiers is sufficient in the light of the response characteristics of the detectors to cover maximum and minimum concentrations anticipated with a maximum signal output of no more than the specified 5 volts.

Reliability is an extremely important aspect of any automatically operated equipment. We have found that the detector system used in this phase of the work is extremely reliable. For example, if the apparatus is opened to the atmosphere for a prolonged period of time, only some 5 minutes of purging is required with reassembly to obtain the same response and stability characteristics that initially prevailed. The cross-section

detectors designed by Melpar have been found to have a reliability exceeding that of the proven workhorse of gas chromatography, the thermal conductivity detector.

5. GAS CHROMATOGRAPHIC OVEN

5.1 Introduction

The major portion of this month's effort on the oven has been directed towards achieving the minimum mass-to-charge ratio in the chemical thermitic ($\text{Al-Fe}_2\text{O}_3$) heaters while still containing the reaction with firing. These heaters have been mounted around the periphery of the oven, the oven has been cooled to 200°K , (dry ice temperature) and then the heaters have been fired sequentially in bringing the oven to 310°K . The oven has been maintained at this temperature in the 200°K environment for over 1 hour. Two ovens which contained simulated valves, columns, and detectors were used in these experiments.

Added temperature stability for the oven has been achieved through the use of $\text{Na}_2\text{HPO}_4 \cdot 12 \text{H}_2\text{O}$ as a heat sink. This salt was added to the dead space within the oven. It has a melting point near 130°K and a heat of fusion of 67 cal/gm. The presence of this salt fulfills two functions: first, since it can soak up heat by melting when the temperature of 310°K is reached, it minimizes temperature overshoot; secondly, the melted salt serves as a good heat reservoir for maintaining this temperature as heat is lost from the oven to the environment.

The feasibility of the chemical heating system for raising the temperature of the oven from ambient to the vicinity of 300°K has been shown. This unit will be demonstrated.

5.2 Experimental Procedure

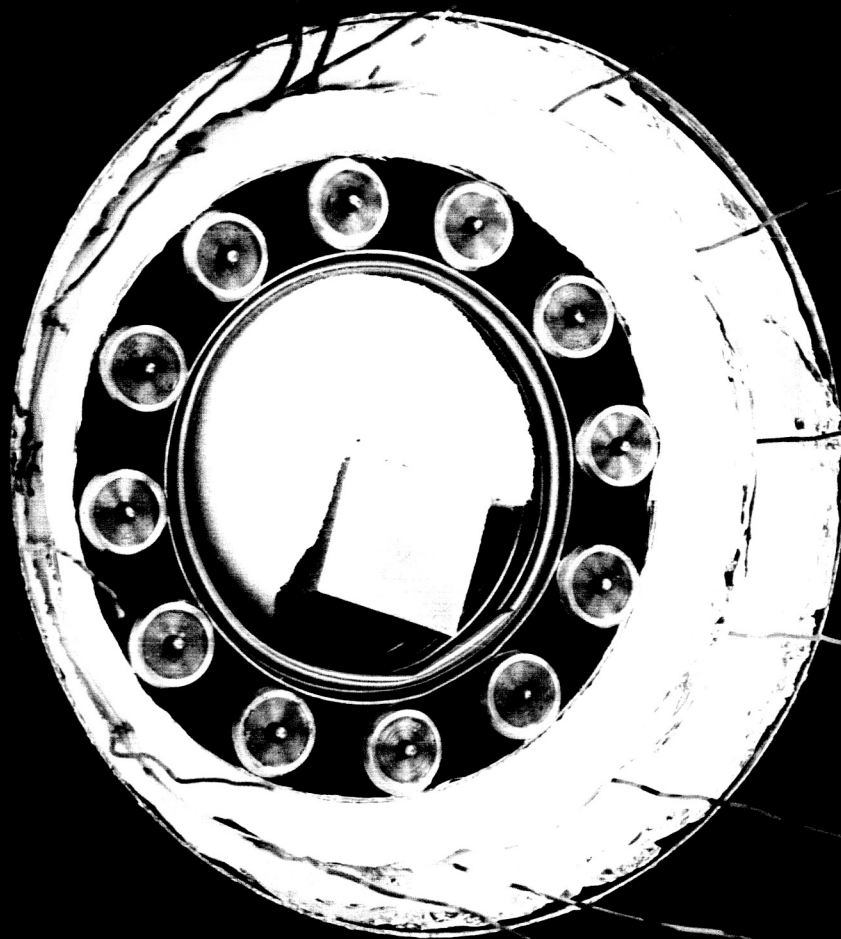
Further studies were made in connection with the containment of the ferricoxide-aluminum thermitic reaction. Containment was quite good when

the wall thickness of the aluminum cartridge in which the charge resided was at least 1/8-inch, and when the container was lined with 1/16-inch ceramic coating. However, even here some failures did occur with firing. These failures were manifest in a melting of a spot within the container and in some cases as a blowout which occurred through the electrical feed-through holes.

In attempting to realize more effective containment, the rate of the reaction was decreased by increasing the size of the ferric oxide particles used. Although it was found that the reaction rate could be appropriately reduced by this means for firing and containment at room temperature, it was also observed that the reaction under these conditions could not be initiated by firing at 200°K.

In the light of the difficulties with the thermitic reaction, it has been decided to use a less energetic one in realizing a greater degree of reliability with firing. Melpar has obtained a chemical mixture used by Atlas Chemical Company of Wilmington, Delaware, in their delay line squibs for the chemical heating. The heat of the reaction of this mixture is about 300 calories per gram. The reliability of containment with these squibs fired at 200°K has been quite good and no failures have been noted. Some 12 of these squibs are sufficient for heating the oven from 200°K to 310°K and for maintaining the oven in the 200°K environment for 1 hour.

The experimental oven construction is shown in the photographs of figure 8; it consists of a 3-inch diameter chamber, 3 inches deep, with 12 chemical heating units mounted around its periphery. The chamber plus heating units are surrounded by 3/4-inch foam insulation. The chemical

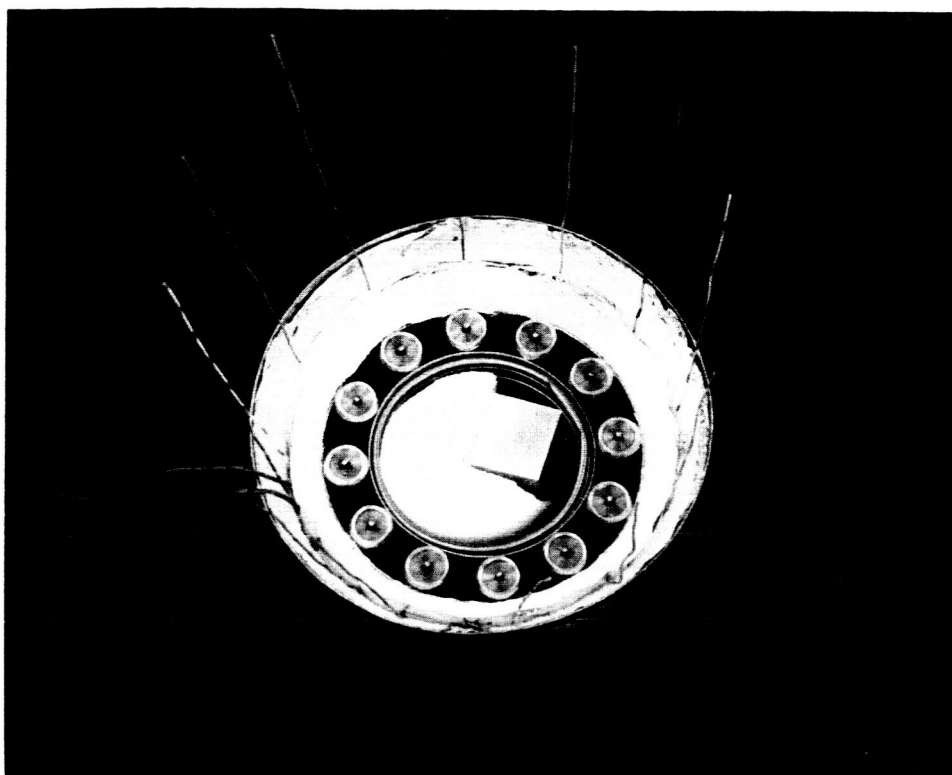




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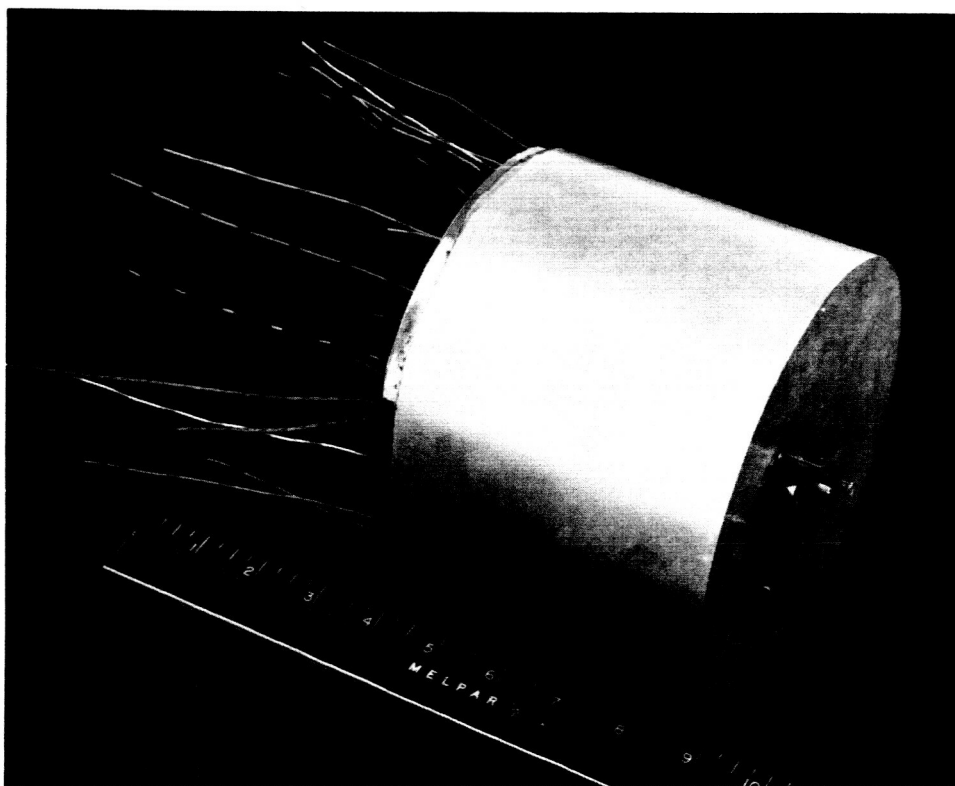


Figure 8. Photographs of Gas Chromatographic Oven

heaters were fired in 2-minute intervals in one experiment and at 1-minute intervals in another experiment. The oven temperature was raised from 200°K to 310°K in a 200°K temperature environment.

5.3 Results

Eight of the chemical heating cartridges of figure 8 are sufficient to bring the temperature of the oven from 200°K to 310°K with an outside temperature of 200°K. The total heat input to effect this temperature rise is 48,000 calories. With the present insulation sheath and the salt heat sink, the oven is maintained at this temperature for approximately 1 hour without additional heat input from any source.

It is to be noted that eight cartridges are sufficient for heating the oven as indicated whether these cartridges be charged the aluminum-iron oxide thermite or the Atlas chemical. In realizing an energy output of about 6,000 calories per cartridge, the same cartridge weight is required for either type charge. It is possible to pack two to three times as much of the Atlas chemical into a given cartridge container as thermitic material and still maintain reliable firing with containment. The use of the chemical heaters charged with the Atlas chemical is quite feasible, and the units can be fired with a high degree of reliability.

The total weight of the oven of figure 8 including $\text{Na}_2\text{HPO}_4 \cdot 12 \text{H}_2\text{O}$, simulated columns, valves, and detectors is only 2.7 pounds. The chemical cartridges may be fired electrically at 3 volts with a current drain of 1/2 amperes (1.5 watts).

The actual sequencing and firing of the chemical charges in their aluminum containers is of course of prime concern. Since the starting

temperature is unknown but may fall anywhere within the range of 140° to 300°K, the heating system must be flexible enough to allow for any ambient temperature within this range in raising the oven to 310°K. (The temperature of 310°K could just as easily be 300°K, the present temperature used for the column operation.) Sequence firing through a thermostatically controlled commutator switch is to be used in realizing this flexibility. A control system of this type has been constructed, and it will be demonstrated with the oven proper.

5.4 Conclusions

The work during the final month of this contract has resulted in the demonstration of the feasibility of chemical heating for the chromatography oven to be used in conjunction with the Mars Gas Chromotography Instrument.

6. ELECTRONIC CONSIDERATIONS

As in other aspects of this program, the major emphasis has been given to the integration of the electronic components with the detectors and other portions of the gas chromatographic system. The electrometers with their automatic zero suppression circuits previously described in past monthly reports have functioned without difficulty. Minor changes in the circuitry were made in adapting the electrometers to the series column system.

6.1 Electrometer Amplifiers

Additional experimental work was carried out on high-input impedance amplifiers and the technique of impedance multiplication by signal sampling. The latter approach appears to offer the greatest promise. The sampling amplifier appears superior in both drift and noise to that required for the gas chromatographic unit. Thus, for the laboratory model, it was decided to use a commercially available sampling-type amplifier.

The concentration range with which one has to contend on this program is 10^5 for nitrogen. The change in current output for this range at the moment is between 10^2 and 10^3 . The present amplifiers are sufficient to handle this range without having to resort to logarithmic amplifiers. However, it may be desired in the encoding to use a logarithmic amplifier in realizing maximum resolution in response data.

6.2 Feedback Amplifier

The feedback amplification system used for automatic zero suppression including the amplifiers, buckout resistor, and the resistive elements of the low pass filter have been mounted on a plugboard which plugs into the

chassis adjacent to its associated electrometer amplifier. The original feedback circuit described in previous reports is being used with the exception of some minor changes in resistor values.

6.3 Logarithmic Amplifiers

At the moment, the two logarithmic amplifiers constructed for this application are not being used. The detector response itself is sufficiently logarithmic. These amplifiers are mounted on plugboards adjacent to the electrometers. They may be easily connected into the circuit if desired.

6.4 Programmers

For the demonstration mechanical programmers will be used. Two programmers are envisioned. One will be required for the sequence firing of the chemical heaters in bringing the oven to a temperature near 300°K. A second more general programmer will be used for controlling the energizing of the electronics, the firing of squibs, and so on.

6.5 Power Supplies

The plus and minus 10-volt regulated power supply shown in figure 29 in the second monthly letter report was found to be adequate. However, with the logarithmic amplifiers in the circuit, the dissipation rating of one of the transistors is too low. If the decision is made to use this amplifier, some minor modifications will have to be made in the power supply.

For the moment, the power supply for the detectors is in the form of 45-volt batteries. The drain on these at a base current of only about 10^{-8} amperes is very small. Due consideration is being given to the use of multivibrator converters as power supplies.

6.6 Packaging

All electronic components, with the exception of the programmers and power supplies, have been mounted into a single package suitable for demonstration. The package with dimensions of $5\frac{1}{2} \times 4\frac{1}{2} \times 8\frac{1}{2}$ inches (height, width, and depth, respectively) is fairly compact. However, no real attempts have been made at miniaturization of the package since some degree of accessibility was desired during the integration period. The two electrometer amplifiers are mounted at opposite ends of the chassis while their respective feedback amplifiers, the voltage regulator, and the voltage connections for the buckout currents were mounted on fiberglass laminate plugboards.

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